Comminuted Meat Emulsions: Differential Thermal Analysis of Fat Transitions

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SUMMARY

Differential thermal analysis (DTA) was employed in analyzing raw materials with a special interest in any relation of fat melting to emulsion stability. DTA curves were obtained on fats in beef and pork materials which had been chilled to 3°C, or frozen at −19°C, and either rapidly or slowly thawed. DTA curves were also obtained on emulsions comminuted to 3°, 10°, 18.5°, 24.5°, 29.5° and 38°C; and the stability of these emulsions was determined on cooking to 68°C.

Data obtained from DTA curves indicated that there were two primary ranges of melting in the beef and pork fats, whether in the raw materials or in the raw and cooked emulsions. The ranges were from 3° to 14°C and from 18° to 30°C for beef fats, and 8° to 14° and 18° to 30°C for pork fats. The instability of emulsions comminuted to more than 18.5°C coincided with the onset of melting of the high-melting portions of fats.

INTRODUCTION

A major problem in manufacturing emulsion-type sausage products, such as frankfurters and bologna, is the tendency of fat to separate during processing. A sausage batter or mix constitutes, at least in part, a form of an emulsion. The various constituents of the sausage batter—water, fat and protein—correspond to the typical continuous and dispersed phases and to the emulsifying agent respectively, of emulsions.

Preparing sausage emulsions generally consists of adding meat to a chopper along with curing agents, seasonings, and water in the form of ice. Water and salt, aided by the action of the chopper knives, solubilize some of the proteins, those in the salt soluble

group being the more effective (Swift et al., 1961). At this point, fat is added and dispersed during chopping. A stable dispersion of fat is achieved when fat is adequately enveloped in protein. If all goes well, both fat and water are bound when the product is subsequently heat-processed. However, there are times when fat and water separate, resulting in what is known as "emulsion breakdown."

Temperature control during emulsification is essential; temperatures customarily are held to 60°F (15.5°C) or lower. Maximum internal temperatures attained in products during cooking and smoking must also be controlled to prevent emulsion breakdown; temperatures approximating 152–156°F (66.6–68.8°C) are used. In addition, precautions are taken with respect to the condition of materials, grinders and choppers. However, the basic causal factors for emulsion breakdown are not understood. A thorough understanding could be the basis for developing means to eliminate poor quality emulsions and losses occurring in reprocessing unstable products, to permit more latitude in the use of meat raw materials and to improve designs of equipment and methods.

Little information is available in the literature concerning the role of fat and the effect of its transitions on the stability of sausage emulsions. One method for improving our understanding of the melting characteristics of typical meat fats and the transitions in fat forms which may occur in making sausage emulsions is to examine meat fats by Differential Thermal Analysis (DTA).

This method has been applied for some time in other areas of fat analysis (Haighton et al., 1958; Hannewijk

et al., 1958; Mares, 1965; and Perron et al., 1961) and can be especially valuable when applied to the present problem. In this study, DTA was employed in analyzing fats in raw materials and emulsions, and the data used to study the relation of fat melting to emulsion stability.

EXPERIMENTAL MATERIALS AND METHODS

Raw materials. The fat and meat samples were taken from commercial grade cow beef and market weight hog carcasses which had been stored at 3°C for 72 hr.

Tissue fat for DTA. Beef fat samples were taken from the round, flank and chuck areas. Pork fat samples were taken from the ham, belly and back fat areas. Prior to analysis, representative samples were stored at 3°C, −19°C, and −19°C and then thawed and held at 3°C for 2 days.

Ground beef and ground pork for DTA. Samples of lean beef and regular pork trimmings, approximately ground twice through a 3/16 in.-plate. 50% lean and 50% fat, were each Samples were analyzed after storage as described previously.

Materials used in emulsions. The remaining portions of the beef carcasses were separated into lean beef and beef trimmings with the pork carcasses being subdivided and regular pork trimmings produced. The lean beef and pork trimmings used in preparing the emulsions were divided into three lots and handled as follows: Lot I, meats chilled at 3°C; Lot II, meats frozen at −19°C and thawed overnight to 0°C; Lot III, meats frozen at −19°C, thawed and held for 2 days at 3°C.

Procedure for DTA. A DuPont 900 Differential Thermal Analyzer was used on approximately 110 mg samples placed in 4 × 28 mm glass tubes and the temperature programmed at 10°/min. In all cases the temperatures critical in the respective determinations, either 3° or −19°C for the samples of fatty tissue and ground beef and ground pork; the temperatures 3°, 10°, 18.5°, 24°, 29.5° or 38°C attained during comminution, and the temperature of 3°C used for storage after cooking, were carefully maintained until the samples were subjected to thermal analysis.

The samples of fatty tissue and ground meat were held in a metal block adjusted to 3°C or to −19°C, when transported from the cooler or freezer to the laboratory. Samples of emulsion when removed from the silent cutter were immediately placed in jars that had been adjusted to appropriate temperatures and then into DTA sample tubes. These were stored in closable vials that were submerged in Dewar flasks containing water at the temperature desired. DTA curves were obtained on heating from the temperature desired. DTA curves were obtained on heating from the temperature at which sample was submitted to 60°C, and on reheating these samples to 60°C after they had been quickly cooled to 0°C. All samples were run in triplicate.

Preparation of emulsions. The basic sausage formula for each batch consisted of 2.25 lb of lean beef, 2.75 lb of pork trimmings, 1.25 lb of flaked ice plus seasoning, cure, sugar and salt.

The lean beef was ground through a ⅝-in. and then through a ³⁄₁₆-in. grinder plate. The pork trimmings were ground twice through a ½-in. plate to obtain uniform mixing of the fat and lean. The lean beef, salt, cure and seasoning plus one-half of the ice were chopped for 2 min in a silent cutter equipped so that the bowl rotated within a stationary jacket. This permitted heating or cooling the emulsion as desired by circulating water at controlled temperatures through the annular space between the bowl and jacket. The ground pork and the remainder of the ice were added and chopping continued.

Samples were taken for DTA and emulsion stability tests when the chopping temperature had risen from 1.5°C to 3°C (8 min); 10°C (11 min); 18.5°C (15 min) and from 1.5°C to 24°C (10 min); 29.5°C (13 min) and 38°C (16 min). In chopping the latter emulsion the temperatures were obtained more rapidly than would be ordinarily possible by using an infrared lamp to increase the ambient temperature and by circulating warm water through the jacket surrounding the chopping bowl.

Emulsion stability test. A stability test was developed in which raw emulsion was placed in a hand stuffer and 34 g samples of emulsion were stuffed into each of three ⅞ × 4 in. polycarbonate tubes. The tubes were stoppered, placed in a 48.8°C water bath and the temperature intermittently raised until the internal temperature of the product reached 68.8°C in about 1.25 to 1.5 hr. One tube in each batch cooked was equipped with a thermocouple and temperature was recorded during cooking.

Samples of emulsion cooked in triplicate were removed from the water bath and the liquid released during cooking immediately decanted into graduated 15 ml tubes. The total volume of liquid, volume of fat, gel-water and proteinaceous solids released during cooking were determined. Averages of triplicate determinations were calculated and are reported as volume in ml per 100 g emulsion. The cooked samples were allowed to cool over night. Emulsion samples which had been comminuted to 10°C and 38°C were wrapped in Saran film and held in a 3°C cooler for 1 week before thermal analysis.

RESULTS

DTA measures the difference in temperature between a sample and an inert reference (glass beads) when both are heated at a uniform rate. Temperature changes that take place within the sample are due to gain or loss of energy as produced by phase transitions. In the present work only approximations were made of the amounts or portions of fats represented by the respective peaks.

In general, the DTA curves indicate that there are two primary ranges of melting in heating pork tissue fat, with melting commencing at 8–9° and extending to 13–14°C and commencing again at 18–19° and extending to 28–30°C. The low melting represents a considerably smaller portion than the high melting. Fig. 1 shows DTA curves obtained on samples of ham tissue fat which had either been chilled to 3°C (1A), frozen at −19°C (1B), or frozen at −19°C, thawed and held at 3°C for 2 days (1C). The DTA curve shown in Fig. 1A indicates melting occurred in chilled ham tissue fat from 8 to 13°C and from 19 to 28°C. The curves in Fig. 1B and 1C indicate that freezing or freeze-thawing had no discernible effect on the melting characteristics of this fat.

The data in Table 1 show the melting characteristics of pork adipose tissue from the back and belly areas, as well as the ham areas. The results indicate a medium sized peak in the low melting range for chilled ham and belly fat, and a smaller one for chilled back fat. Freezing or freeze-thawing affected the pattern of melting in the low range in the case of the ham and belly fats, but not the back fat. The results also indicate a medium sized peak in the high range in melting belly tissue fat and a larger one in this range for the ham and back-fat tissue fat. There were no discernible changes in these melting characteristics on freezing or freeze-thawing.
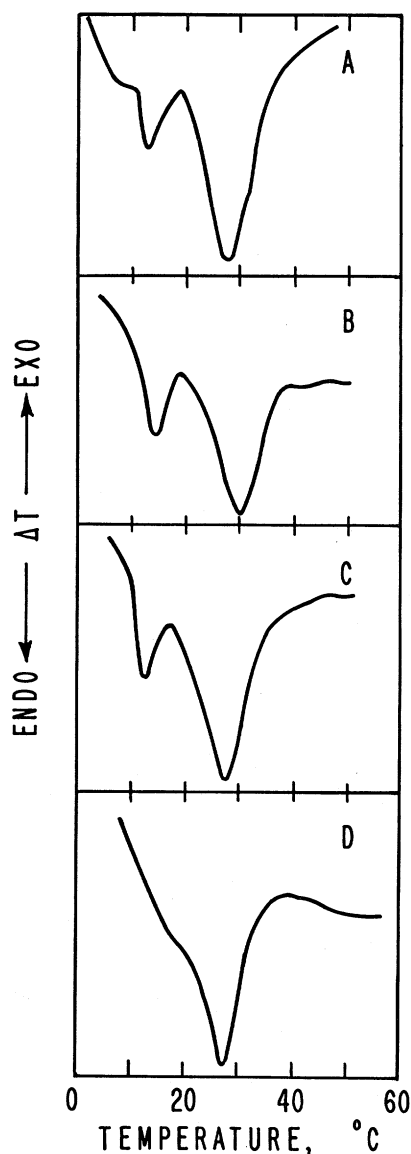

Fig. 1. DTA curves of pork ham tissue fat: A—Chilled to 3°C; B—Frozen at −19°C; C—Frozen at −19°C, thawed and held at 3°C for 2 days; D—Reheated A.

The DTA curve shown in Fig. 1D was obtained on a ham tissue fat sample which had been heated to 60°C, immediately cooled to 0°C and reheated to 60°C. This quick-cooling and immediate reheating was of value in determining possible transitions in crystallization that could result. The data indicate essentially one large melting range extending from 5° to 28°C. Hence, on cooling after the initial heating during DTA, the two characteristic melting ranges normally found in natural tissue fats did not immediately reappear.

The DTA curves indicate that beef adipose tissue melted in two primary ranges, a small to medium portion melting from 3° to 14° and a small–medium to large portion of the fat which began to melt at 18° and was completely melted at 30°C. Fig. 2 shows DTA curves obtained on beef flank tissue fat which had been handled in the same manner as the pork tissue fats. The DTA curve shown in Fig. 2A indicates melting occurred in chilled flank tissue fat from 3° to 10.5° and from 13° to 24.5°C. The DTA curves in Figs. 2B and 2C indicate that freezing and freeze-thawing, in contrast to results obtained with pork fats, did have some effect on the melting characteristics of beef fat.

The data in Table 2 indicate the melting characteristics of beef adipose

tissue from the round and chuck, in addition to the flank areas. The results show that there were differences in the melting of each fat in chilled form and that this melting was modified by freezing or freeze-thawing. The difference in the magnitude of melting in the low-melting range was decreased or eliminated by freezing.

The three fats differed in the amounts melting in the high-melting range. These amounts were most marked between the round and either the flank or chuck fats. For example, that from the round had a small and that from the chuck a large broad zone of melting in the high range. On freezing or freeze-thawing the amount of high-melting fat present in the round and chuck fats was unchanged, while the amount in the flank fat was appreciably decreased.

The DTA curve in Fig. 2D was obtained on analysis of beef flank tissue fat which was heated, quickly cooled and then reheated. A difference in the effect of this treatment on the character of the DTA curves as obtained on reheating pork and beef tissue fat samples can be observed by comparing the curves shown in Figs. 1D and 2D. The results indicate that the melting characteristics of the fats depend on time-temperature conditions. Similar shifts in the melting of fats caused by temperature treatments have been re-

ported by Hannewijk *et al.* (1958) who employed DTA.

Fig. 3A and B show DTA curves obtained on the ground lean beef and ground pork trimmings, respectively, that were used in the preparation of emulsions. The DTA curve in Fig. 3A indicates that the fat present in the
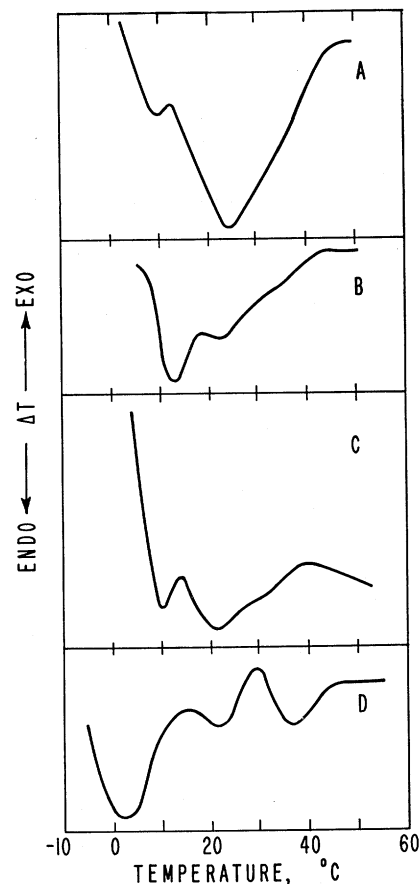

Fig. 2. DTA curves of beef flank tissue fat: A—Chilled to 3°C; B—Frozen at −19°C; C—Frozen at −19°C, thawed and held at 3°C for 2 days; D—Reheated A.
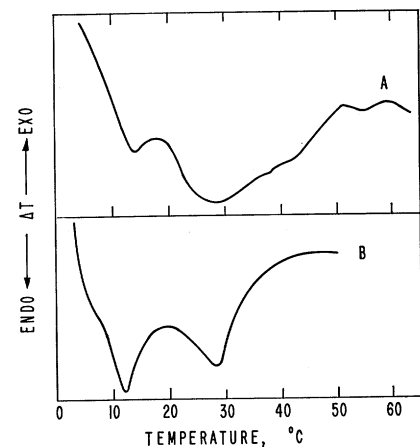
Table 1. The melting characteristics of pork adipose tissue.

| Tissue | Treatment | Melting ranges | | | |
| | | 8–14°C | | 18–30°C | |
		S–M[1]	M	M–L	L
Ham	Chilled		+		+
Back	Chilled	+			+
Belly	Chilled		+	+	
Ham	Frozen	+			+
Back	Frozen	+			+
Belly	Frozen	+		+	
Ham	Frozen-thawed	+			+
Back	Frozen-thawed	+			+
Belly	Frozen-thawed	+		+	

[1] Approximate size of peak. S—small; M—medium; L—large.

Table 2. The melting characteristics of beef adipose tissue.

| Tissue | Treatment | Melting ranges | | | | | |
| | | 3–14°C | | | 15–30°C | | |
		VS[1]	S	M	S	M	LB
Round	Chilled			+	+		
Flank	Chilled		+				+
Chuck	Chilled	+					+
Round	Frozen			+	+		
Flank	Frozen			+	+		
Chuck	Frozen			+			+
Round	Frozen-thawed		+		+		
Flank	Frozen-thawed		+			+	
Chuck	Frozen-thawed	+					+

[1] Approximate size of peak. VS—very small; S—small; M—medium; LB—large broad.

Fig. 3. DTA curves of chilled (3°C) ground tissue: A—Lean beef and B—Regular pork trimmings.

Table 3. Effect of temperature of emulsification on the amounts of components released on cooking to 68.8°C.[1] Volume of components released, ml/100 g emulsion.

Maximum temp. during emulsification	Fat			Gel			Solids			Total volume		
	Chilled	Frozen	Frozen-thawed	Chilled	Frozen	Frozen-thawed	Chilled	Frozen	Frozen-thawed	Chilled	Frozen	Frozen-thawed
°C												
3	0.0 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.4 ± 0.1	1.7 ± 0.3	3.9 ± 1.02	0.0 ± 0.0	0.1 ± 0.1	0.2 ± 0.0	0.4 ± 0.1	2.1 ± 0.3	4.0 ± 0.4
10	0.0 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	1.1 ± 0.2	2.8 ± 0.5	5.8 ± 0.5	0.0 ± 0.0	0.2 ± 0.1	0.3 ± 0.0	1.1 ± 0.2	3.3 ± 0.6	5.9 ± 0.5
18.5	0.5 ± 0.1	0.4 ± 0.1	0.6 ± 0.0	1.9 ± 0.2	4.3 ± 0.7	9.9 ± 1.5	0.0 ± 0.0	0.5 ± 0.1	0.4 ± 0.1	2.4 ± 0.4	5.2 ± 0.8	11.1 ± 1.0
24.0	3.6 ± 0.4	0.6 ± 0.0	3.8 ± 0.2	14.3 ± 0.1	10.0 ± 0.6	19.2 ± 1.2	7.4 ± 0.3	0.9 ± 0.2	2.8 ± 0.1	25.0 ± 0.4	11.5 ± 0.7	25.8 ± 1.3
29.5	6.2 ± 0.4	5.7 ± 0.1	10.9 ± 0.3	11.1 ± 0.7	12.7 ± 0.8	11.0 ± 0.6	14.2 ± 0.9	14.7 ± 0.7	18.9 ± 0.3	31.6 ± 0.6	33.2 ± 0.5	40.7 ± 0.8
38.0	8.0 ± 0.1	6.9 ± 0.1	11.4 ± 0.3	11.9 ± 0.7	15.1 ± 0.4	14.9 ± 0.5	14.1 ± 1.1	11.2 ± 1.2	14.0 ± 1.1	33.5 ± 0.4	33.3 ± 1.2	41.2 ± 0.4

[1] Average value of three samples tested ± standard deviation of the mean.

chilled ground beef samples contained a small portion melting from 4° to 13°C, as in the other beef fats analyzed. The high-melting fat melted over a broad range from 17° to 28°C, as in the case of the beef fats previously discussed; however, the amount melted was low when contrasted with that melted in beef flank fat as shown in Fig. 2A.

The DTA curve in Fig. 3B for the fat in ground regular pork trimmings shows that melting occurred in two zones, 6° to 13° and 18° to 29°C, as in the other pork fats analyzed. However, the amounts of fat melting in these zones were approximately equal, whereas the high melting fat represented a much larger proportion of the fat in the samples of belly, ham and back fats. Reproducible DTA curves were not obtained on the frozen ground beef and pork samples due to interference from the melting of ice at the lower temperatures.

The results obtained in investigating fat melting in relation to the stability of emulsions chopped to different maximum temperatures are shown in Table 3 and Fig. 4. As indicated by measurements of the stability of emulsions on cooking, using the release of fat, gel or solids as criteria; chopping at temperatures higher than 18.5°C, i.e., 24.0°, 29.5° or 38°; produced marked evidence of emulsion breakdown. Measurements of release of fat, gel, solids, or total volume were of substantially equal value as criteria.

The nature of the solids fraction was not determined; however, its characterization could yield significant knowledge of the mechanism involved. The data show that emulsions prepared from frozen materials were as stable as those prepared from chilled materials at corresponding temperatures, while those prepared from frozen-thawed materials were somewhat less so. However, increasing chopping temperatures produced instability in all three in a similar pattern.

The melting characteristics of the fats in the emulsions and the stability of emulsions are shown in relation to melting and chopping temperatures by two curves in Fig. 4. Curve A was obtained by DTA of an emulsion chopped only to 3°C. Curves also were obtained for emulsions chopped to 10°, 18.5°, 24.0°, 29.5 and 38°C. Curve B shows the volume of fat released on cooking emulsions that were produced by chopping to the above temperatures. As indicated by curve B, accelerated breakdown occurred on chopping to higher than 18.5°C which is the approximate temperature at which high melting fat should commence to melt, as indicated by curve A.

The curves obtained by DTA on emulsions chopped to 10°, 18.5°, 24°, 29.5° and 38°C showed that melting occurred as chopping temperatures increased approximately, but not exactly to the extent that would be predicted from inspection of Curve B. Fat in the emulsions chopped to these temperatures was found to have melted at least to the extent that would be expected, i.e., the emulsion chopped to 18.5°C contained no fat melting at a lower temperature and, within the limits of sensitivity of the method, fat was completely melted when 29.5°C had been attained during chopping.

DTA curves for emulsions chopped to 10°C showed no zone of melting from 8 or 9° to 15°C, seemingly indicating that more melting occurred than could be accounted for if 10°C was actually the maximum temperature attained. A reasonable explanation would be that localized heating occurred in the emulsion in immediate contact with the cutting surfaces of the chopper. If so, provided a technique were available, this is a factor which might profitably be assessed with respect to fat melting.

A corresponding situation has been studied by Thornburg et al. (1957)—a zone of melting which was produced through heat development in sectioning fresh-frozen tissue with microtomes. DTA on samples of cooked emulsion, after 1 week of storage at 3°C, showed that the solid fats melted in two ranges, 8° to 15° and from 18° to 29°C, similar to uncooked emulsions chopped to 3°C. However, while curves showed
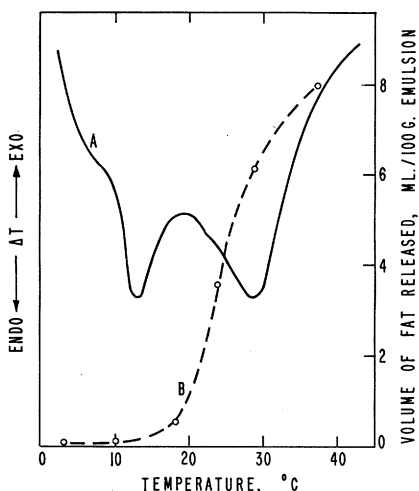

Fig. 4. The relation of chopping temperature and fat melting to emulsion stability. Curve A—DTA curve for emulsion chopped to 3°C; Curve B—Fat released on cooking emulsions which had been chopped to 3°, 10°, 18.5°, 24.0°, 29.5° and 38°C.

that the zones were approximately of equal size in analysis of uncooked samples, the high-melting portion was considerably larger in DTA curves obtained on cooked samples, possibly indicating that a new pattern of mixed crystals had formed on cooling.

DISCUSSION

Sausage makers have learned that meat emulsions produced by chopping beyond certain temperatures tend towards instability and have tended towards caution; for example, a temperature of 55°F (12.7°C) in a maximum recommended for use in commercial establishments in a text published by AMI (1953).

In emulsions prepared by Hansen (1960), some instability was noted in emulsions chopped to 72°F (22.2°C) while it was marked on chopping to 81°F (27.2°C); in emulsions prepared by Helmer et al. (1963) at 70°F (21.1°C) some breakdown also occurred on cooking. The present results are essentially in agreement with those the authors cited, indicating that chopping temperatures in excess of 18.5°C produce unstable emulsions using standard materials and operations.

It has been indicated by Shannon (1966), presumably based on industrial experience, that temperatures as high as 70–72°F could be used if the fat in emulsion products were mainly beef fats and they were immediately smoked. This suggests that fat melting may be implicated in the effects of increasing temperature, for example, as inferred in Anon. (1961).

The results of tests relating chopping temperatures to emulsion stability showed that chopping higher than 64.4° to 66.2°F (18° to 19°C) produced unstable emulsions. Melting of the high-melting fats also began at this temperature; however, this melting alone need not be responsible for the onset of instability.

Alternate explanations of the phenomena suggest themselves. One is that emulsification of the low-melting fat exhausted the emulsifying capacity of the meats, consequently melting that produced additional fat liquefaction produced instability. Another is that demands on proteins were markedly greater in emulsification in the critical temperature range, due primarily to temperature, in which case the presence of additional fat melted at 18–19°C or higher temperatures was not directly responsible for emulsion instability.

Some support exists or can be deduced for either alternative, in fact, further research may show both to be valid. The fact that beef fat can apparently be chopped to higher temperatures than the lower melting pork fats, as previously noted, suggests that the proportion of fats that will become liquefied determines stability on chopping to increasing temperatures. On the other hand, temperature rise *per se* will linearly decrease the capacity of meat proteins to emulsify oil (Swift *et al.*, 1961).

Accounting for the instability of emulsions chopped at higher temperatures could involve the proteins, which are the principal emulsifying agent. Investigators have speculated that as the temperature rises during chopping, protein may become partially denatured and incapable of stabilizing the fat dispersion, thus permitting separation during smoking and cooking. Meat proteins are not normally unduly temperature sensitive below 70°F (21.1°C). Meat proteins must be heated higher than 40°C to produce marked heat denaturation (Bate-Smith, 1935) and retain considerable stability at 20°C. Consequently, if protein breakdown is accelerated on moderately increasing temperatures of emulsification, it appears that it is markedly supplemented by a concurrent event such as one involving the fat.

Since the present results do not justify a selection from the alternatives, a further assessment of the contribution of fat characteristics and of temperature in studies involving the emulsification of liquid and solid fractions of beef and pork fats is in progress. In addition, other investigations are needed to determine the extent and effect of localized heating on fat melting and emulsion stability.

DTA of meat tissue fats showed that they melt in two zones, one at low and the other at higher temperatures. In fats containing a number of different glycerides as meat fats do, the number of melting patterns possible is unpredictable, varying with tendencies to form either or both mixed crystals and polymorphic forms. This depends on temperatures and temperature variations, as indicated by Bailey (1950). His data showed samples of beef fat melting in six segments and lard in five as determined in dilatometric measurements. Therefore, the melting patterns of meat tissue fats are comparatively simple.

An indication of the complexity possible is shown in the melting of reheated quick-chilled beef fats. The observation that the melting of frozen or frozen-thawed fats was substantially the same as that of chilled fats is significant since industrial practice frequently involves such treatments. Based on these data, behavior on chopping would be essentially alike.

The data show discernible differences between fats according to the location of fats in each species. The differences were mainly in the proportions of fats melting in the characteristic ranges, rather than in differences in temperature ranges.

If the interaction of temperatures with fat characteristics significantly affects emulsion stability, fats from different locations affect the maximum temperature at which stable emulsions can be produced. Similarly the smaller low melting zone and the larger high melting zone of beef fats as compared with pork fats could be the basis for differences in permissible temperatures for emulsification.

Additional investigations under way may determine the importance of the differences in fat melting characteristics that have been observed, beginning with clarification of the specific events leading to emulsion breakdown.

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